Using powder characterisation methods to assess blending behaviour

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Blending a mixture of powders to homogeneity is a critical step in many manufacturing processes. Making uniform tablets, for example, relies on the homogeneous dispersion of active ingredients, often in small quantities, with excipients such as binders and disintegrants to form a suitable feed blend for the press. Similarly, in industries as diverse as food production and metal powder processing, homogeneity is crucial to defining product characteristics: flavour or colour in foods for instance, or the mechanical properties of a sintered metal component.

Collective understanding of the factors that influence blending kinetics is still in its infancy with relatively little information in the public domain. Furthermore, from a practical viewpoint there is little advice on how to characterise individual powders, or a mixture, so as to predict blending performance.

Here we consider the informational requirements needed to achieve better blending, and highlight the potential value of dynamic powder testing in this context. Correlations of positron emission tomography (PET) data with flow energy measurements suggest that dynamic data may be a reliable predictor of blending behaviour and consequently useful for the development and optimisation of blending processes.

Informational requirements for optimised blending

The knowledge base that underpins effective blending is not yet secure, despite the technique's widespread use. For example, there is only limited understanding of the powder characteristics that influence how quickly and easily a multi-component mixture will blend to homogeneity, a critical issue when designing and optimising a formulation. In addition, the optimal analytical toolkit to support blending has yet to be assembled. Individual powders can be difficult to reliably characterise, blends even more so, and real-time monitoring is a considerable challenge.

Therefore progress towards better blending practice relies, to a significant extent, on identifying analytical techniques that are appropriate for the task. One requirement is for powder characterisation techniques that enable the prediction of blending performance, and which allow researchers to determine how different powders, or mixtures, will perform in a blender. Such methods would help improve our understanding of blending processes, promote the selection of appropriate feed materials (where there is choice), and potentially streamline scale-up. The complementary need is for process analysers that enable continuous monitoring of blenders within the plant environment.

Current industrial practice typically involves thief sampling, the taking of between 10 and 30 samples from different points within the blender for analysis in the laboratory. This technique is widely used during development and into manufacturing. So, for example, in a batch blending process the blender might be operated for a certain period of time and then sampled to determine whether composition is consistent across the range of sample points.

There are two major drawbacks with this approach. Firstly the sampling process itself disturbs the bed and secondly a very large number of samples are needed to reach just a single assessment of homogeneity. Thief sampling and work-up can be a time-consuming and lengthy process, ill-suited to supporting effective decision making in the manufacturing environment.

On-line measurement is of growing interest and here attention has increasingly turned to the application of spectroscopic techniques (Near Infrared and Raman) and other methods such as Laser Induced Fluorescence (LIF) and thermal effusivity. These technologies can all be used to effect continuous measurement within the process environment but usually at just a single point, potentially compromising their ability to support full optimisation. It is usual to keep QC back-up in place to provide an assurance of success, and the issue of method validation, usually by thief sampling, remains.

Introducing positron emission tomography

Positron emission tomography (PET) is a nuclear imaging technique with its roots in medical scanning. It creates images by detecting pairs of gamma rays released by a positron-emitting tracer. In blending it can therefore be used to track the dispersion of a tracer to a defined degree of homogeneity, and to evaluate the effect of certain parameters on that blending process. Clearly the technique is not currently well matched to industrial requirements but it does offer the opportunity to access reliable data that can be correlated with, for example, powder testing data to usefully identify parameters that are relevant to the performance of blending processes.

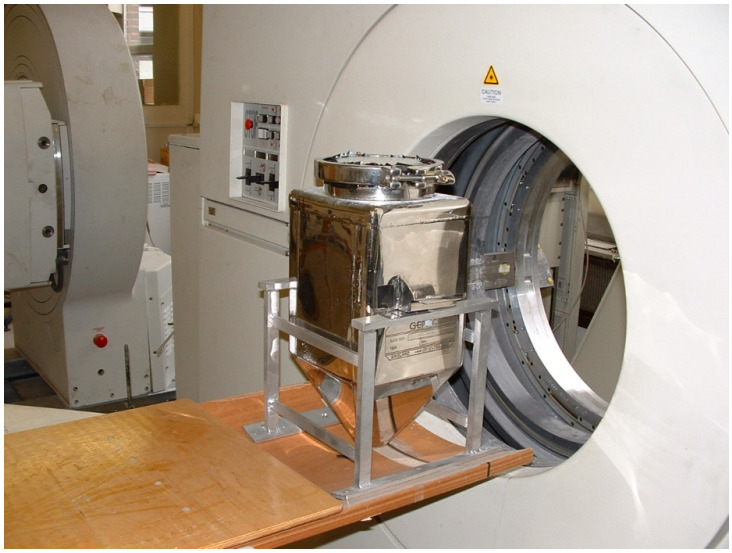

Figure 1: A mixing vessel entering the PET camera to enable measurement of the extent of homogeneity within the blend

PET is a non-invasive technique capable of characterising an entire blended mass through a stainless steel vessel wall[1]. Its application changes neither the contents of the blender nor the structure of the powder bed so it provides a true assessment of the state of blend without disturbing it in any way. Figure 1 was taken during a blending experiment. The bin contains the powders being blended, which include a small bolus (1 -2% by mass) of a compatible radioactive material, in this case microcrystalline cellulose (MCC). After being tumbled for a number of revolutions, the bin is transferred to the camera to evaluate the degree of dispersion of the radioactive particles. It is then returned to the blender and each step is repeated to the point of acceptable homogeneity (see figure 2).

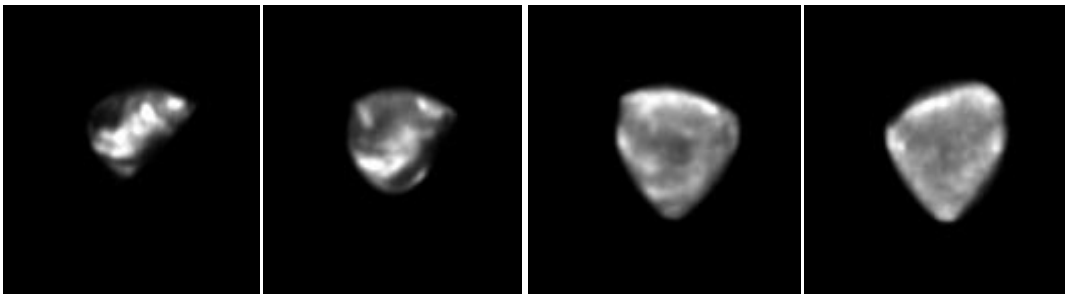

Figure 2: Tracking the blending process through to homogeneous dispersion of the radioactive tracer – images gathered after 5, 20, 55 and 100 revs

The disadvantages of PET are that it requires the use of a radioactive tracer and can, at the moment, only be applied on the laboratory scale. Only one component is tracked and analysis is relatively slow, although comparable to the rates achieved with thief sampling. On the other hand, PET enables detailed scrutiny of the blended mass in a way that gives new and highly reliable insight into blending.

In this set-up for example, each scan produces an image consisting of 62 slices of the volume of the vessel with approximately 10^6 data points produced in total, per analysis. Each data point represents a very small volume of the whole mass, around 43 mm^3. In the resulting images, the light intensity in each region relates directly to the number of radioactive counts emanating from it i.e. the amount of tracer present. Thanks to its high resolution, the technique offers the important benefits of being able to track low concentrations of tracer (because of the sensitivity of the radioactive detector) and examine in detail areas where poor mixing is suspected.

Using PET to identify powder characteristics relevant to blending

Using PET a series of simple blending experiments were conducted to assess the impact of defined variables on process performance and to identify powder properties that correlated with the behaviour observed.

In a first step, a 2% radioactive bolus of MCC was blended into a five litre volume of MCC, see figure 3. The PET data relating to blend homogeneity are represented by the parameter %RSD, with lower %RSDs associated with greater homogeneity; below 5% the blend can be considered effectively homogeneous. Duplicating the experiment at rotation speeds of 10 and 15 rpm enabled assessment of the impact on blender rotation speed on process performance.

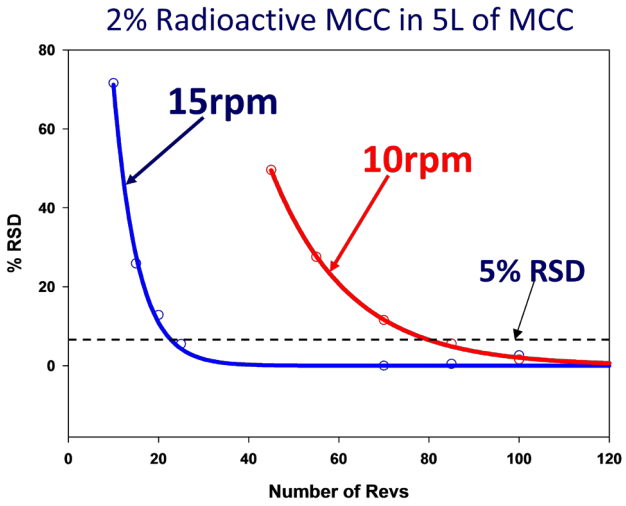

Figure 3: With MCC higher blending speeds lead to faster blending; fewer revolutions are required to reach homogeneity

In this experiment higher blender speeds are associated with faster blending, a result entirely consistent with expectations. However, repeating the experiment with a different excipient, sodium benzoate, produced quite different results (see figure 4). Here lower blender rotation speeds accelerate the blending process.

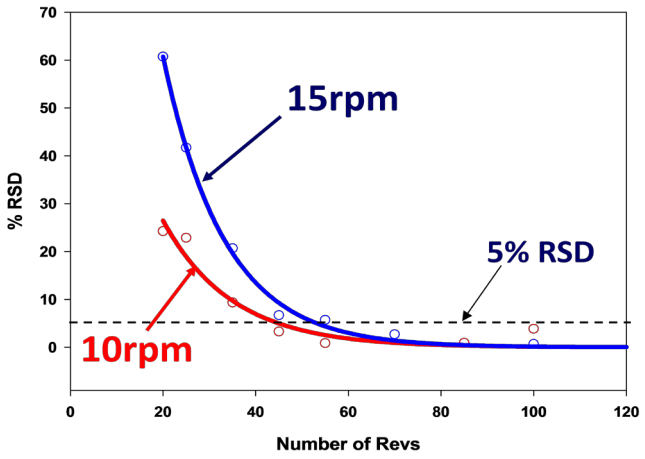

Figure 4: With sodium benzoate higher blender speeds are detrimental to processing performance; more revolutions are required to achieve blend homogeneity

Both MCC and sodium benzoate were characterised using the FT4 Powder Rheometer (Freeman Technology, UK)[2] as well as a number of other routine powder testing techniques. This was to determine whether any easily measured powder properties could be correlated with this observed behaviour. The FT4 offers shear, bulk and dynamic powder property measurement, and dynamic parameters are especially relevant in describing powder flow and mixing.

Dynamic powder characterisation involves precisely measuring the axial force and torque acting on a helical blade as it rotates down through a sample along a defined path. These measurements are used to generate flow energy values that define the amount of energy needed to induce powder flow. By varying the speed of rotation of the blade it is possible to quantify how the powder responds when induced to move more quickly or more slowly. Such behaviour is directly relevant in assessing the impact of blender speed. Figure 5 shows flow energy data for the MCC and sodium benzoate.

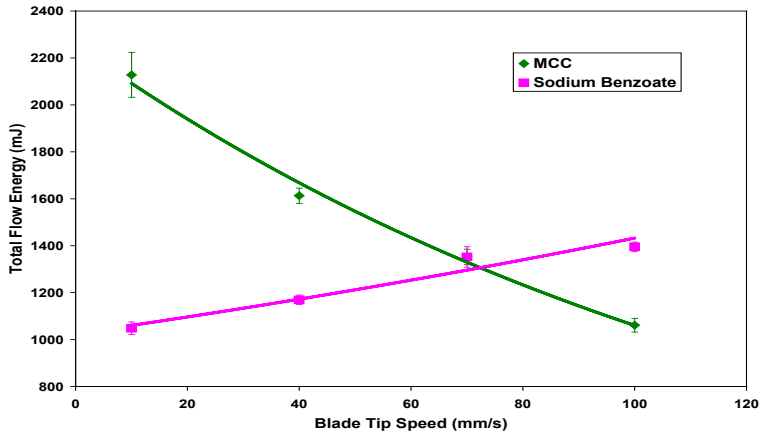

Figure 5: Dynamic powder test data show that the MCC flows more easily when induced to flow more quickly while the sodium benzoate exhibits the opposite trend

In general, powders flow more easily at higher flow rates. In dynamic testing, flow energy tends therefore to reduce in line with increasing blade tip speed, as it does here with the MCC. However, with the sodium benzoate flow energy increases with increasing blade tip speed indicating greater resistance to flow, as flow rate increases. These data therefore directly correlate with the observed trends in blending performance.

In fact, the sodium benzoate and MCC are morphologically dissimilar. The MCC has spherical particles. Increasing blender speeds, or indeed blade tip speeds, causes these particles to become more aerated so they flow and mix more easily. The sodium benzoate particles on the other hand, are platelet shaped, and simply lock together more enthusiastically at higher flow rates. They experience translational /rotational frustration which simply gets worse at higher blender speeds, inhibiting effective dispersion. This trend was captured by dynamic flow energy data but not by any other powder property such as angle of repose, shear cell data, flow through an orifice, tapped density methods or particle size.

Investigating multi-component blends

To further assess the reliability of correlations between flow energy data and blending performance the experimental study was extended to a ternary system. A bolus of 2% radioactive MCC was loaded into the blender on top of a layer of two litres of MCC, which in turn had been layered on top of three litres of lactose. Figure 6 shows flow energy data, for the lactose, MCC and the binary mixture and, alongside it, the PET data recorded during blending.

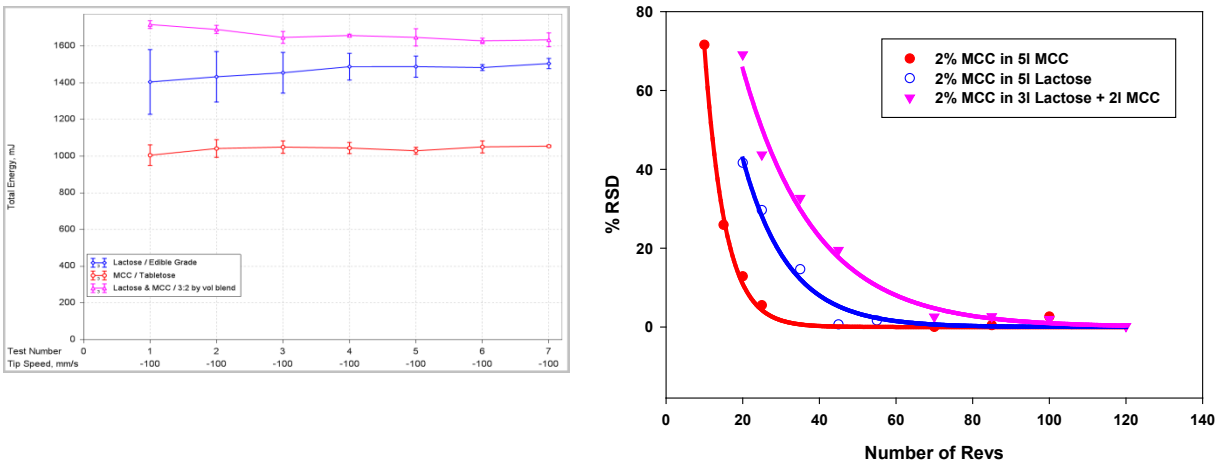

Figure 6: Dynamic flow energy data for a mixture of lactose and MCC predicts the unexpected result that MCC will blend less easily into a lactose/MCC mixture than into either individual component

These results show that the traced MCC blends more easily into bulk MCC than it does into an identical volume of lactose. Also that, while it might be expected that MCC/lactose mixture results would lie somewhere between these extremes, the mixture is associated with the worst blending performance. This result could be attributed to preferential partitioning of the MCC into a like material but the flow energy data suggests that the poor dispersion is instead directly linked to an increased resistance to flow of the bulk MCC/lactose mixture, which inhibits particle movement and dispersion. These results therefore reinforce the finding that the correlation between flow energy and blending time is a robust one.

Looking ahead

Although techniques such as PET are clearly some way from being an accessible tool for industry they bring value by facilitating the identification of readily available characterisation methods that can be used to optimize blending processes. The experimental work carried out here demonstrates that dynamic powder characterisation can support this goal, by providing data that enable the prediction of blending performance.

Looking ahead, results such as these point to opportunities to correlate critical aspects of blending performance, such as blending time and blender speed, with powder properties, for reliable process simulation. This is an approach already being pursued by leading researchers in the field who are looking at how to predict blend characteristics from the properties of constituent components, as well as, focusing more widely on the simulation of blending processes. Into the future powder testing may therefore play an important role in reducing current reliance on expensive, large scale blending trials.

References

[1] Seville, J. P. K., Parker, D. J. & Ingram, A., 2005, "Probing Processes Using Positrons", Chemical Engineering Research & Design, 83, A7, pp788-793
[2] Freeman, R. E., 2007, "Measuring the flow properties of consolidated, conditioned and aerated powders – A comparative study using a powder rheometer and a rotational shear cell", Powder Technology, 174, 1-2, pp 25-33

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